

# Ex. 5

**C•T•F•A Compendium of Cosmetic Ingredient Composition**

# ***Specifications***

## ***Editors***

***Joanne M. Nikitakis  
Gerald N. McEwen, Jr., Ph.D., J.D.***

## ***Editorial Advisor***

***Alfred G. Wich***

***The Cosmetic, Toiletry and Fragrance Association  
1110 Vermont Avenue, N.W., Washington, D.C. 20005***

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1110 Vermont Avenue, N.W., Washington, D.C. 20005

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MS 00029

J&J-0039079

C.T.F.A. Specification**TALC**

**DEFINITION:** Talc is an essentially white, odorless, fine powder which is ground from naturally occurring rock ore. It consists of a minimum of 90% hydrated magnesium silicate, with the remainder consisting of naturally associated minerals such as calcite, chlorite, dolomite, kaolin, and magnesite, and containing no detectable fibrous, asbestos minerals.

TEST	SPECIFICATION	METHOD
Color .....	As specified by the buyer and showing no change after heating	Heat 1 to 2 g at 200°C for 5 minutes.
Odor .....	As specified by the buyer	
Identification .....	1. Close match to CTFA spectrum-IR with no indication of foreign materials or 2. (Alternate) Close match to X-ray Powder Diffraction File No. 19-770, published by ASTM, showing the most intense reflections at $d$ values about 9.35, 1.53, and 4.95 Å	CTFA G 3-1
Slip .....	As specified by the buyer	
Lustre .....	As specified by the buyer	
Water-Soluble Iron .....	Passes test	USP (Current)
Water-Soluble Substances ....	0.1% maximum	USP (Current) (Reaction and Soluble Substances)
Acid-Soluble Substances .....	As specified by the buyer 6.0% maximum	CTFA E 32-1
Screen Test .....	100% through 100 mesh 98% minimum through 200 mesh Finer grades: as specified by the buyer	CTFA C 6-1
Loss on Ignition .....	6.0% maximum	CTFA E 36-1
Arsenic (as As) .....	3 ppm maximum	CTFA E 1-1, Parts I-A and II
Lead (as Pb) .....	20 ppm maximum	CTFA E 2-2, Parts I-A and II
Fibrous Amphibole (Asbestiform Tremolite et al.) ..	None detected	CTFA J 4-1
Free Crystalline Silica (Quartz) .	As specified by the buyer	CTFA J 5-1 (DTA) Alternate: CTFA J 6-1 (X-ray)
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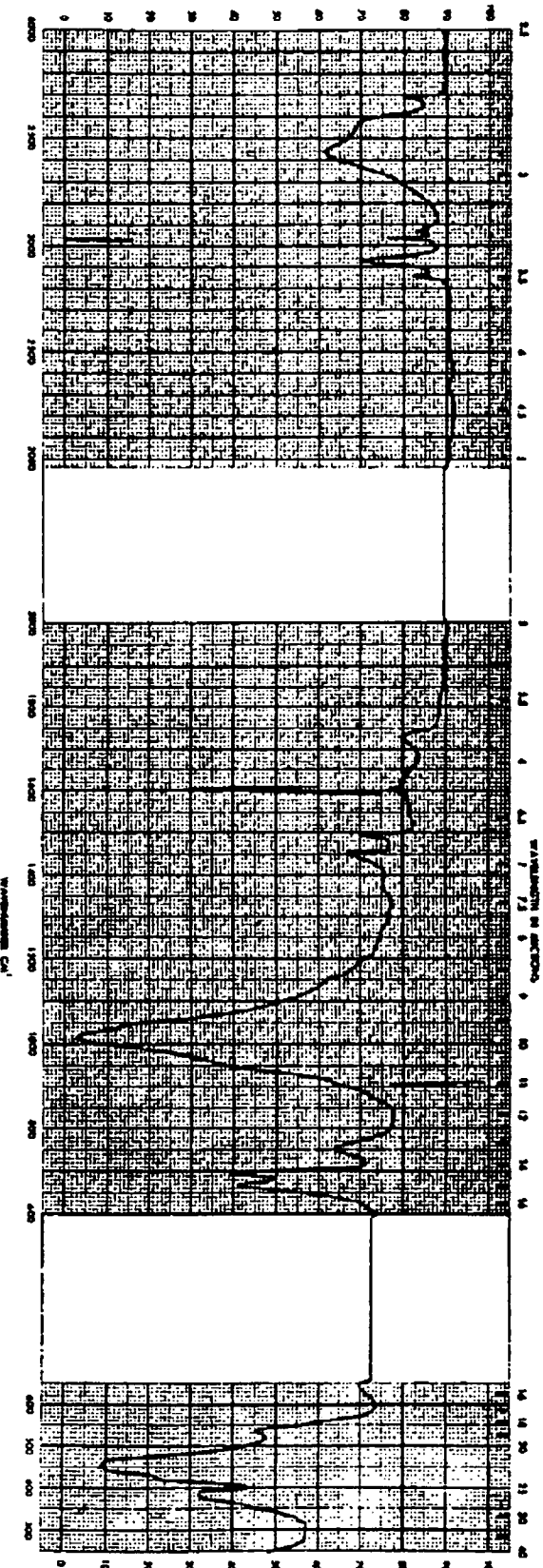
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TALC

C-T-F-A Specification

MS 00031

J&J-0039081



Product: Talc  
Phase: Solid  
Method: Pressed Pellet, 0.19% In KBr  
Reference: Air  
Instrument: Beckman IR-10  
Optics: Grating

This spectrum is presented for identification purposes only and is not intended as a standard of purity.

## ***Preparation of Infrared Spectra*** ***(Pressed Pellet Technique)***

### **Principle**

The sample is mixed with an alkali halide, the mixture pressed into a thin, transparent disc, or pellet, and the infrared spectrum of the pressed pellet recorded.

### **Apparatus**

1. Evacuatable die with plunger and barrel, designed for pressing out a thin disc or pellet (Note 1)
2. Hydraulic laboratory press, capable of delivering 24,000 lbs total load
3. Spectrophotometer, infrared, recording, capable of covering the same range shown on the specific CTFA spectrum of interest
4. Mortar with pestle, agate or mullite, 75 mm o.d.
5. Wig-L-Bug Amalgamator (Crescent Dental Mfg. Co., Chicago, Illinois) equipped with timer and stainless steel vial and balls
6. Vacuum pump, capable of attaining pressures less than 2 cm Hg

### **Reagents**

1. Potassium bromide, or other alkali halide (KI, KCl, CsBr, or TlBr) as given for the specific CTFA spectrum of interest; ACS reagent of a spectroscopic grade

### **Procedure**

Place about one mg of the sample in the clean dry mortar and add approximately 200 mg of the specified alkali halide (Note 2). Grind the mixture for one-half to one minute to produce a uniform powder with an even distribution of sample throughout the alkali halide, and with the particle size of the sample as small and as uniform as possible (Note 3). Use the Wig-L-Bug Amalgamator in conjunction with the grinding operation, either before or after adding the alkali halide, or use it instead of the mortar grinding if desired (Note 4).

Transfer the ground mixture to the evacuatable die, distributing the powder evenly on plunger face or within powder cavity. Assemble die according to the manufacturer's instructions.

Place the assembled die in the press, apply 50 to 100 lbs pressure, and evacuate using vacuum pump for about two minutes at less than 2 cm Hg. Increase the pressure to 20,000 to 24,000 lbs total load and maintain for two minutes.

Carefully break the vacuum and slowly release the pressure. Carefully remove the thin, transparent, pellet from the die and place in a suitable holder.

Record the infrared spectrum covering the same range shown on the CTFA spectrum.

### **Notes**

1. The small wrench-operated presses, consisting of a one-unit press body and suitable stainless steel bolts, may be used instead of the heavier evacuatable die and hydraulic press, provided that acceptable pellets can be produced.

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C•T•F•A Method G 3-1

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2. The concentration of sample in alkali halide should normally be from 0.1% to 0.5%. When more intense spectra are desired, it is preferable to increase the pellet thickness rather than to increase the concentration of the sample in the alkali halide.
3. Additions of volatile solvents including dry ice may be used to aid in the grinding operation if desired.
4. Place about 200 mg of the sample-halide mixture into a stainless steel vial, add one stainless steel ball, cap securely, and mix on the Wig-L-Bug for two to five minutes.

**Reference**

"Instructions—Evacuatable Potassium Bromide Die," Manual 990-9487, Revised July 1966, Perkin-Elmer Corp., Norwalk, Conn.

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## Acid Soluble Substances in Talc

### Principle

A sample of talc is mixed with diluted hydrochloric acid and the insoluble portion is removed by centrifugation and filtration. The filtrate is sulfated, evaporated to dryness and ignited. The acid soluble matter is determined gravimetrically as the sulfate.

### Apparatus

1. Controlled temperature water bath
2. Beaker, Griffin, 100 ml
3. Volumetric flask, 50 ml
4. Centrifuge
5. Centrifuge tubes (2), 50 ml
6. Graduated Cylinder, 50 ml
7. Sintered glass funnel, ultra fine porosity, 60 ml (0.9 to 1.4  $\mu$ m) Coming #36060-UF or equivalent
8. Vacuum flask, 125 ml
9. Pipette, 10 ml
10. Platinum or Vycor evaporating dish or equivalent

### Reagents

1. Hydrochloric Acid, diluted, 10% v/v
2. Sulfuric Acid, diluted, 10% v/v

### Procedure

Weigh 2 g ( $\pm 0.001$ ) of sample into a 100 ml beaker, add 40 ml diluted hydrochloric acid, and place in water bath at 55 °C ( $\pm 2.5$  °), with occasional stirring for 30 minutes. Immediately remove beaker from bath and pour contents into a 50 ml centrifuge tube, rinsing beaker and stirring bar with 2 to 3 ml of distilled water. Centrifuge at 5000 RPM for 30 minutes (Note).

Filter supernatant through ultra fine porosity sintered glass funnel into vacuum flask, using 2 to 3 ml of distilled water to rinse the centrifuge tube and funnel, and using care to avoid dislodging the packed talc at the bottom of the tube. Filtrate must be clear (Note). Pour filtrate into 50 ml volumetric flask, rinsing with 3 to 5 ml distilled water. Adjust to volume with distilled water.

Pipette 25 ml of adjusted filtrate into tared, ignited evaporating dish. Add 2 ml of diluted sulfuric acid and evaporate to dryness.

Ignite at 800 °C ( $\pm 25$  °) for 1 hour. Cool and weigh.

### Calculation

$$\% \text{ Acid Soluble Substances} = \frac{\text{Weight of Residue in g} \times 200}{\text{Weight of Sample in g}}$$

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C•T•F•A Method E 32-1

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**Note**

Examine the centrifuged sample carefully to assure good clarity before proceeding with filtration. If haziness is detected, centrifuge the sample for an additional 15 to 30 minutes as necessary.

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C-T-F-A Method C 6-1

## **Screen Test for Fine Powders**

### **(Wet Screening Method)**

#### **Principle**

The fine particles of the sample are washed through the sieve. The larger particles are retained on the sieve, and dried and weighed.

#### **Apparatus**

1. Wire-cloth sieves, US Standard, 8-in (203 mm); conforming to the specifications given in ASTM Designation E 11-70
2. Weighing dishes
3. Camel hair brush, small

#### **Reagent**

Alcohol, 95%, SD 3A or equivalent

#### **Procedure**

Weigh a 10.00 g ( $\pm 0.01$  g) sample of material and quantitatively transfer to the required sieve (Note 1). Use a separate 10 g sample for each sieve required.

Attach a thin flexible piece of clean rubber tubing about two feet long with an I.d. of about  $\frac{1}{4}$ " to a water faucet, and turn on water very slowly until a thin stream is flowing through the tubing.

Wash the sample through the sieve by directing a stream of water upon it from the rubber tubing (Note 2). Alternately tip the sieve from one side to the other, washing the sample from the higher level of the sieve to the lower. Continue the washing until satisfied that all the fine material has been washed through. A small camel hair brush may be used to facilitate washing and to break up lumps; however, care must be taken not to break or fracture particles that are larger than screen openings, either by use of the camel hair brush or by too forceful a stream of water.

Dry the screen and contents on a steam bath, and then place in a 105 ° C oven for 30 min. When dry, carefully brush the residue into a small tared weighing dish and weigh to the nearest 0.001 g.

#### **Calculations**

$$\% \text{ Residue Held on Screen} = \frac{\text{g of Residue} \times 100}{\text{g of Sample}}$$

$$\% \text{ Material through Screen} = 100.0 - \% \text{ Held on Screen}$$

Report results to one decimal place.

#### **Notes**

1. Carefully examine each sieve each time it is used to make sure that no cracks or holes have developed.
2. For materials that are not wet by water and that are *insoluble in alcohol*, a stream of alcohol from a wash bottle may be used to wash the sample through the sieve. After thoroughly wetting with alcohol, the washing of most materials can be completed with water. The complete determination may be done with alcohol exclusively if necessary.

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**Reference**

ASTM Designation: D 1514-60, Sieve Residue from Carbon Black

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J&J-0039087

C.T.F.A Method E 36-1

## Ash or Loss on Ignition of Talc

### Principle

The sample is ignited at about 825 ° C for four hours and the residue determined gravimetrically.

### Apparatus

1. Platinum dish or crucible
2. Electric muffle furnace
3. Desiccator

### Reagents

1. Drierite, indicating (Calcium Sulfate, Anhydrous), four to eight mesh, desiccant

### Procedure

Ignite a platinum dish in a muffle furnace at 825 ° C ( $\pm 25$  ° C) for one hour. Cool to room temperature in a desiccator and weigh to the nearest mg for the tare weight.

Accurately weigh to the nearest mg one gram of sample into a tared platinum dish. Place the dish into a muffle furnace and ignite at 825 ° C ( $\pm 25$  ° C) for four hours. Cool to room temperature in a desiccator and weigh to the nearest mg.

### Calculations

$$\% \text{ Ash} = \frac{W \times 100}{G}$$

OR

$$\% \text{ Loss on Ignition} = 100 - \% \text{ Ash}$$

Where: W = weight of ash in grams  
G = weight of sample in grams

Report results to one decimal place.

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MS 00038

J&J-0039088

## **Arsenic Determination**

### **(Silver Diethyldithiocarbamate Method)**

The complete analytical procedure for the determination of trace amounts of arsenic by the *Silver Diethyldithiocarbamate Method* is presented in two parts: *Part I, Sample Preparation*, and *Part II, Arsine Evolution and Measurement*. The *Sample Preparation* varies according to the nature of the material being tested, and different sample preparations are given as *Parts I-A, I-B, I-C*, etc. The *Arsine Evolution and Measurement* is relatively standard, and in practice directly follows any one of the different sample preparations.

#### **Part I-A: Sample Preparation—Acid Soluble Arsenic in Talca, Pigments, and Most Acid Insoluble Materials**

##### **Principle**

The sample is extracted with hot dilute acid to obtain the acid soluble arsenic.

##### **Apparatus**

1. Beaker, Griffin, 250 ml, 50 ml
2. Filter paper, Whatman No. 4 or equivalent
3. Volumetric flask, 100 ml
4. Pipette, 50 ml
5. Erlenmeyer flask, Pyrex, 125 ml,  $\frac{1}{4}$  24/40 G.G. joint

##### **Reagents**

1. Hydrochloric acid, 0.5 N
  2. Distilled water
- (All reagents must have low arsenic content.)

##### **Procedure**

All glassware must be scrupulously clean. Wash with hot dilute solution of sulfuric acid. Rinse thoroughly with distilled water and then with acetone. Dry in an electric or steam-heated oven.

Weigh a 5.00 g ( $\pm 0.01$  g) sample directly into a 250 ml beaker. Add 50 ml of 0.5 N HCl (Note 1), and mix thoroughly. Also prepare a blank and carry through all steps of the procedure so as to have available for subsequent use:

Bring to a boil on a low hot-plate, and boil gently for 15 minutes, taking care that the mixture does not splatter or foam excessively. Stir and mix two or three times during the boiling. Let the undissolved material settle.

While still hot, decant supernatant extract through Whatman No. 4 filter paper into a 100 ml volumetric flask, retaining as much as possible of the insoluble material in the beaker (Note 2). Add 10 to 15 ml of hot 0.5 N HCl to the beaker and slurry mixture. Heat again to a boil on the hot-plate. Let settle and decant through same filter paper; collect washings in the flask with extract. Extract 3 to 4 times with 10 to 15 ml of hot 0.5 N HCl.

Cool flask and contents to 25 ° C and make up to volume with additional 0.5 N HCL

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C·T·F·A Method F 1-1

PART I-A

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Pipette a 50.0 ml aliquot of the prepared solution into a 125 ml Erlenmeyer flask. Cover with an inverted 50 ml beaker. If smaller aliquots of the solution are used, in anticipation of high amounts of arsenic in the sample, make up the difference to 50 ml with 0.5 N HCl.

Proceed directly with Part II, continuing from "To each 125 ml Erlenmeyer flask (Generator Flask) add . . . ."

Notes

1. For calcium carbonate or chalk with relatively high acid insoluble content, *cautiously* add 20 ml 6 N HCl instead of the 50 ml 0.5 N HCl, then boil and continue with the hot 0.5 N HCl extracts as specified. For calcium carbonate with low acid insoluble content, follow *Sample Preparation, Part I-B*. See Note 5, Part II.
2. When the undissolved material fails to settle, catch the whole insoluble portion on the filter paper, and then return entire portion with paper to the beaker. Add the 10 to 15 ml of hot HCl, macerate, heat with stirring, and refilter through a second filter paper. Repeat for subsequent extracts if necessary. Treat the blank accordingly.

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MS 00040

J&J-0039090

C.T.F.A Method F 1-1

PART II

**Arsenic Determination**  
**(Silver Diethyldithiocarbamate Method)**

The complete analytical procedure for the determination of trace amounts of arsenic by the *Silver Diethyldithiocarbamate Method* is presented in two parts: *Part I, Sample Preparation*, and *Part II, Arsine Evolution and Measurement*. The *Sample Preparation* varies according to the nature of the material being tested, and different sample preparations are given as *Parts I-A, I-B, I-C*, etc. The *Arsine Evolution and Measurement* is relatively standard, and in practice directly follows any one of the different sample preparations.

**Part II: Arsine Evolution and Measurement**

**Principle**

Arsenic (V) is reduced to arsenic (III) with stannous chloride in the presence of iodide ions, and evolved as arsine ( $\text{AsH}_3$ ) by reaction of zinc in sulfuric acid solution. The arsine is absorbed in pyridine solution of silver diethyldithiocarbamate and arsenic determined quantitatively by spectrophotometric measurement of the resulting soluble red complex.

**Apparatus**

1. Arsine Absorption Trap, Pyrex, with  $\text{F 24/40}$  G.G. joint (Note 1)
2. Erlenmeyer flask, Pyrex, 125 ml,  $\text{F 24/40}$  G.G. joint
3. Spectrophotometer: photoelectric, covering a spectral range of 475 nm to 575 nm with wavelength scale readable to 1 nm, and absorbance accurate to within a 1% absolute error
4. Absorption cells, quartz, stoppered, matched pair, 10 mm pathlength
5. Volumetric flasks, 20 ml, 1 liter
6. Beakers, 50 ml, 250 ml
7. Ear syringe, rubber, small
8. Absorbent cotton, bleached

**Reagents**

1. Arsenic trioxide,  $\text{As}_2\text{O}_3$ , Primary Standard, 99.95% to 100.05%, ACS grade
2. Stannous chloride,  $\text{SnCl}_2 \cdot 2\text{H}_2\text{O}$ , ACS grade
3. Potassium iodide, KI, ACS grade
4. Lead acetate,  $\text{Pb}(\text{C}_2\text{H}_3\text{O}_2)_2 \cdot 3\text{H}_2\text{O}$ , ACS grade
5. Silver diethyldithiocarbamate (AgDDC),  $\text{AgSCSN}(\text{C}_2\text{H}_5)_2$
6. Zinc, granular, 20 mesh, ACS grade
7. Pyridine,  $\text{C}_5\text{H}_5\text{N}$ , ACS grade
8. Sodium hydroxide solution, dilute, 10%
9. Sulfuric acid solution, dilute, 40% (2 volumes  $\text{H}_2\text{SO}_4$  to 3 volumes distilled water)
10. Hydrochloric acid, 36.5% to 38.0% HCl, ACS grade
11. Stannous chloride solution, 40%  
Dissolve 40 g ( $\pm 1.0$  g) of stannous chloride in 100 ml of concentrated hydrochloric acid. Store in a glass bottle, and use within 3 months.
12. Potassium iodide solution, 15%  
Dissolve 15 g ( $\pm 0.5$  g) of potassium iodide in 100 ml of distilled water. Store in a glass bottle and discard when any brown color appears.

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C•T•F•A Method F 1-1

PART II

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13. Lead acetate-impregnated cotton

Soak absorbent cotton in a saturated solution of lead acetate, squeeze out excess solution, and dry in an air oven at about 100° C. Store in a small wide mouth jar.

14. Silver diethyldithiocarbamate solution, 0.5%

Weigh 1.000 g ( $\pm 0.0005$  g) of silver diethyldithiocarbamate directly into a 250 ml beaker and add about 100 ml of pyridine. Stir with glass rod to dissolve. *Do not heat.* Transfer solution to a 200 ml volumetric flask, rinse beaker, and make up to volume. Store solution in a brown glass bottle at room temperature in the dark (Notes 3 and 4).

(All reagents except the Primary Standard must have low arsenic content.)

**Procedure**

All glassware must be scrupulously clean. Wash with a hot dilute solution of sulfuric acid. Rinse thoroughly with distilled water, and then with acetone. Dry in an electric or steam-heated oven.

Prior to starting the following Arsine Evolution all standard samples, test samples, and blanks must have been prepared previously by one of the various specific *Part I Sample Preparation* procedures, and thus will be in 125 ml Erlenmeyer flasks, covered with inverted beakers. Each flask will contain 50 ml of solution, with pH adjusted when necessary, ready to continue as given herein.

**Arsine Evolution**

To each 125 ml Erlenmeyer flask (Generator Flask) add 10.0 ml ( $\pm 0.5$  ml) of 40% sulfuric acid (Note 5), 2.0 ml ( $\pm 0.5$  ml) of 15% potassium iodide solution, and 10 drops of the 40% stannous chloride solution. Swirl flask gently to mix contents thoroughly. Place on steambath, still covered with the inverted beaker, for 5 minutes ( $\pm 1$  minute).

Remove from heat and cool to 25° to 28° C.

Prepare a clean absorption trap for each flask. Place a loose wad of lead acetate-impregnated cotton in the lower part of the trap so that all evolved gases must pass through it. Lubricate the standard-taper joint tightly with a thin layer of stopcock grease.

For each sample and blank weigh out a separate 3.0 g ( $\pm 0.1$  g) portion of 20 mesh granular zinc.

Measure 5.00 ml ( $\pm 0.01$  ml) of the 0.5% silver diethyldithiocarbamate solution into each arsine absorption trap.

Using a small powder funnel pour a 3.0 g portion of the granular zinc rapidly into the sample solution. Immediately and quickly place a trap on the flask so that no arsine is lost when the zinc starts the evolution of the arsine and hydrogen. Give the trap a few partial turns to be sure it is seated tightly. Check to see that the gases are evolving vigorously, and bubbling smoothly in small numerous bubbles through the absorbing solution.

Run the arsine evolution for 45 minutes ( $\pm 1$  minute) at room temperature, swirling each flask gently a few times after each 10 minute period.

Loosen the trap and raise slightly from 125 ml flask. With an ear syringe gently draw the absorbing solution back and forth through the sintered glass plug and lower portion of the small side tube until there are no dark colored portions of the absorbing solution in or around the lower part of the trap.

Pour the mixed absorbing solution directly from the trap into a clean dry 10 mm absorption cell.

Measure the absorbance of each developed absorbing solution, against a reagent blank (Note 6) that was carried through all the steps of both the preparation procedure and the arsine evolution procedure. Read at the wavelength that was determined from the spectrophotometric curve and that was used in plotting the

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C·T·F·A Method F 1-1

PART II

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Standard Concentration Curve. From the Standard Concentration Curve determine the number of micrograms of arsenic.

Calculations

$$\begin{aligned} \text{Arsenic (as ppm As)} &= \frac{\text{mcg As in Sample}}{\text{Wt of Sample in Grams}} \\ &= \frac{\text{mcg As in Aliquot}}{\text{Wt of Sample in Aliquot}} \end{aligned}$$

Standard Concentration Curve

Weigh 0.660 g ( $\pm 0.0005$  g) of Primary Standard arsenic trioxide ( $\text{As}_2\text{O}_3$ ), dried previously in an oven for 2 hours at  $105^\circ\text{C}$ , directly into a 100 ml beaker. Add 25.0 ml of 10% sodium hydroxide (NaOH) solution. Stir with a glass rod to dissolve and transfer to a 1 liter volumetric flask. Rinse beaker several times, mix thoroughly, adjust to  $25^\circ\text{C}$  ( $\pm 1.0^\circ\text{C}$ ), and fill to the 1 liter mark with distilled water. Label this flask as Standard Stock Solution A. The concentration equals 500 micrograms of Arsenic (as As) per ml.

Make a 1 to 100 dilution from Standard Stock Solution A in a second volumetric flask, and label it Dilute Stock Solution B. The concentration equals 5 micrograms of Arsenic (as As) per ml. Discard this solution when more than 3 days old.

Using appropriate aliquots of Dilute Stock Solution B, prepare a series of 5 to 6 solutions in 125 ml Erlenmeyer flasks, differing in total arsenic content from 2.5 micrograms to 15.0 micrograms. Include a blank. Add 45 ml ( $\pm 2.0$  ml) of distilled water to each flask, and cover with inverted beakers.

Proceed with the Arsine Evolution, as given above, starting from "To each 125 ml Erlenmeyer flask (Generator Flask) add \_\_\_\_\_." Complete through the development of the reddish-brown color and the mixing of the absorbing solutions.

Select one of the developed and mixed solutions, that was prepared with an arsenic content of about 5.0 mcg, and pour it directly from the trap into a clean dry 10 mm cell. Fill the reference cell with the developed reagent blank solution that was carried through all the steps of the arsine evolution procedure, and prepare a spectrophotometric curve as shown in Figure 1.

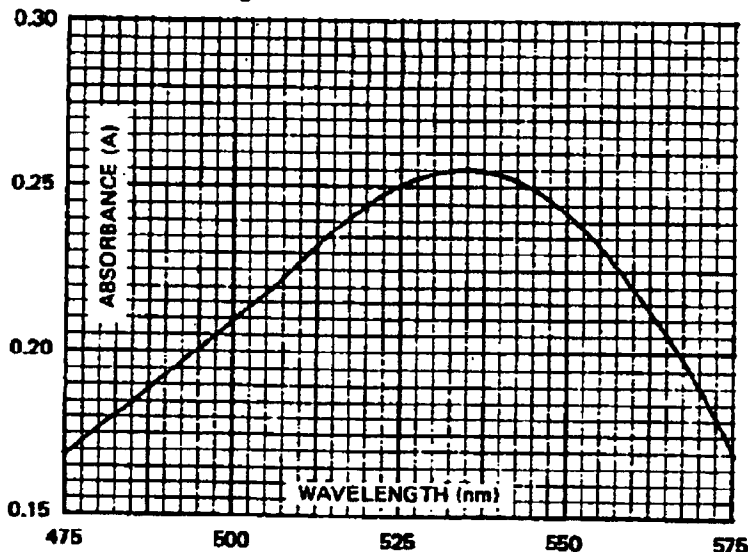


Figure 1

From the curve select the exact wavelength in the 525 to 545 nm area which gives maximum absorbance.

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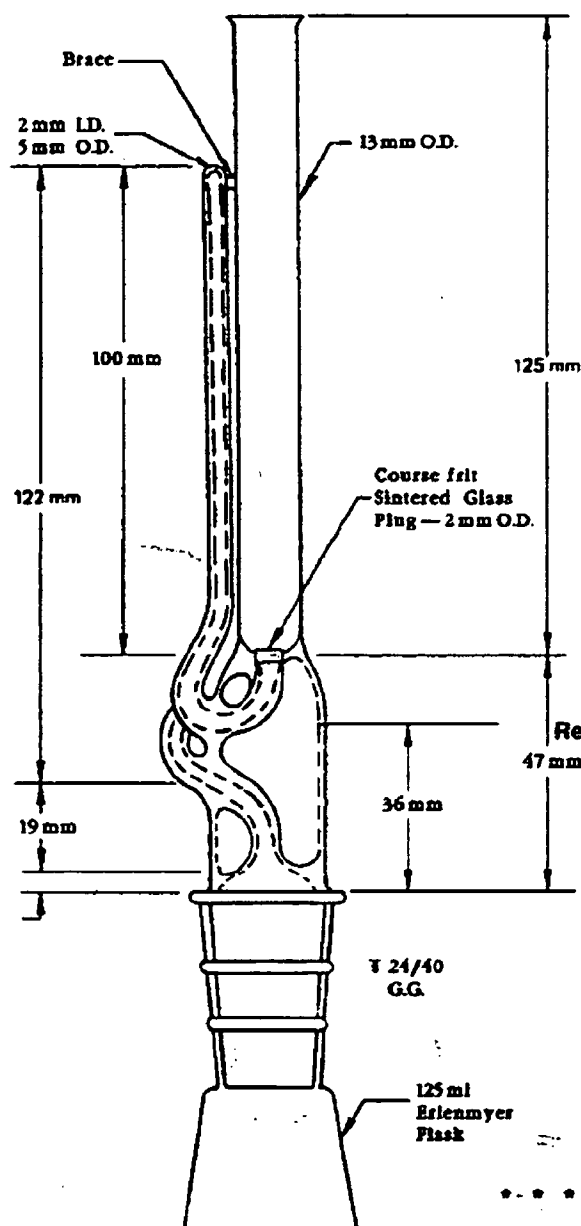
PART II

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Measure the absorbance of each prepared solution at the wavelength of maximum absorbance against the reagent blank. Plot a Standard Concentration Curve.

**Notes**

**1. Arsenic Absorption Trap**



2. Silver diethyldithiocarbamate is a lemon-yellow color when fresh, and usually a light tan after drying. The dry salt is apparently quite stable when stored in a light-resistant container. The decomposed material is pink to brown in color, and often has a strong odor.
3. Do not use any 0.5% silver diethyldithiocarbamate solution that is over one month old, or shows any appreciable darkening. The freshly made solution will normally have an absorbance of about 0.4 to 0.7 at 425 nm in a 10 mm cell when read against distilled water.
4. Prepare a spectrophotometric curve and select the wavelength of maximum absorbance for each different lot of silver diethyldithiocarbamate. Prepare a new standard concentration curve if the selected wavelength varies by more than  $\pm 5$  nm from the previous lot.
5. Use 10 ml of conc hydrochloric acid in lieu of the 10 ml of 40% sulfuric acid when a sample contains calcium, barium, or other cation that gives an insoluble sulfate.
6. Check the Reagent Blank frequently by reading its absorption against the 0.5% AgDDC solution. Check all reagents if it shows an increase in absorbance of 0.050 or more.

**References**

1. Fisher Scientific Company - "Technical Data TD-142," July, 1960
2. Standard Methods of Chemical Analysis, N. Howell Furman, Volume I, Sixth Edition, March, 1962
3. Food Chemicals Codex, First Edition, National Academy of Sciences-National Research Council, 1966

**Figure 2. Arsenic Absorption Trap**

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J&J-0039094

JNJ 000304880

C.T.F.A Method F 2-2

PART I-A

**Lead Determination**  
**(Colorimetric Dithizone Method)**

The complete analytical procedure for the determination of trace amounts of lead by the *Colorimetric Dithizone Method* is presented in two parts: *Part I, Sample Preparation*, and *Part II, Color Development and Measurement*. The *Sample Preparation* varies according to the nature of the material being tested, and different sample preparations are given as *Parts I-A, I-B, I-C*, etc. The *Color Development and Measurement* is relatively standard, and in practice directly follows any one of the different sample preparations.

**Part I-A: Sample Preparation—Acid Soluble Lead in Talcs, Pigments, and Most Acid Insoluble Materials**

**Principle**

The sample is extracted with hot dilute acid to obtain the acid soluble lead.

**Apparatus**

1. Beaker, Griffin, 250 ml
2. Funnel, 75 mm
3. Filter paper, Whatman No. 4
4. Flask, volumetric, 100 ml
5. Hot plate, electrical
6. Watch glass, 90 mm

**Reagents**

1. Hydrochloric acid, 0.5 N
2. Distilled water

**Procedure**

Weigh 10.0 g ( $\pm 0.01$  g) of sample directly into a 250 ml beaker. Add 50.0 ml of 0.5 N hydrochloric acid, cover with a watch glass and bring to a boil on a hot plate.

Boil gently for 15 minutes. Do not allow excessive foaming to occur. Cool and allow the sediment to settle.

Decant the supernatant through Whatman No. 4 filter paper into a 100 ml volumetric flask. Retain as much of the sediment as possible in the beaker. Add 10 ml of hot water to the sediment, mix, allow to settle, and decant through the filter paper into the volumetric flask. Repeat the washing procedure two more times.

Finally, wash the filter with 10 to 15 ml of hot water and collect in the volumetric flask. Cool the volumetric flask to room temperature, make to volume with water and mix well.

Run a blank along with the sample.

Proceed directly with *Part II*.

\* \* \* \* \*

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C•T•F•A Method F 2-2

PART II

**Lead Determination**  
**(Colorimetric Dithizone Method)**

The complete analytical procedure for the determination of trace amounts of lead by the *Colorimetric Dithizone Method* is presented in two parts: *Part I, Sample Preparation*, and *Part II, Color Development and Measurement*. The *Sample Preparation* varies according to the nature of the material being tested, and different sample preparations are given as *Parts I-A, I-B, I-C*, etc. The *Color Development and Measurement* is relatively standard, and in practice directly follows any one of the different sample preparations:

**Part II: Color Development and Measurement**

**Principle**

The lead is complexed with excess dithizone, and the resulting red complex, along with a definite proportion of the excess uncombined green dithizone, are extracted under standard volumetric and pH conditions with chloroform from aqueous solution. The lead is determined quantitatively by spectrophotometric measurement of the red complex.

**Apparatus**

1. Separatory funnels, 250 ml and 500 ml
2. Filter paper, 9 cm
3. Spectrophotometer
4. Absorption cells, 1 cm
5. Funnel, Buchner, 75 mm i.d.
6. Filter paper, Whatman No. 1, 70 mm diameter
7. Filter paper—specially prepared—soak overnight in 1%  $\text{HNO}_3$  and wash with large volumes of distilled water on a Buchner funnel
8. Flask, volumetric, 500 ml
9. Beaker, Griffin, 50 ml

**Reagents**

1. Lead nitrate
2. Nitric acid
3. Chloroform
4. Distilled water
5. Ammonium hydroxide, dilute (1 part + 99 parts  $\text{H}_2\text{O}$ ), metal free
6. Hydrochloric acid
7. Potassium cyanide, phosphate free
8. Ammonium hydroxide

**Lead Standard Solution.** Dissolve 20 to 50 g  $\text{Pb}(\text{NO}_3)_2$  in a minimum volume of hot water and cool with stirring. Filter the crystals, with suction, on a small Buchner funnel, redissolve and recrystallize. Dry the crystals to constant weight at  $105^\circ\text{C}$ . Cool in a desiccator and store in a tightly stoppered bottle. The material has no water of hydration and is not appreciably hygroscopic. Prepare a stock solution containing 2 mg of lead per

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PART II  
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ml in 1% nitric acid ( $2 \text{ mg Pb} = 3.197 \text{ mg Pb(NO}_3)_2$ ). Dilute 5.0 ml of stock solution to 1000 ml with 1% nitric acid to obtain 10 mcg Pb per ml *Lead Standard Solution*.

*Nitric Acid 1%*. Dilute 10 ml of colorless nitric acid (sp gr 1.40) to 1 liter with distilled water.

*Dithizone (diphenylthiocarbazone)*. Dissolve 1.0 g reagent grade dithizone in 75 ml of chloroform and filter if any insoluble material remains. Extract in a separatory funnel four times with 100 ml portions of dilute ammonium hydroxide. Dithizone passes into the aqueous phase to give an orange solution. Filter the aqueous extracts into a large separatory funnel through a cotton pledget inserted in the stem of the filtering funnel. Acidify slightly with hydrochloric acid and extract the precipitated dithizone with three 20 ml portions of chloroform. Combine the chloroform extracts in a separatory funnel and wash three times with distilled water. Draw the chloroform solution into a beaker. Evaporate on a steam bath with gentle heating. Avoid splattering as the solution approaches dryness. Remove the last traces of moisture by heating one hour at  $50^\circ \text{C}$  in vacuo. Store the dry reagent in a dark, tightly stoppered bottle. Prepare reagent solution for the determination to contain 10 mg per liter in freshly distilled chloroform and store in the dark at  $5^\circ$  to  $10^\circ \text{C}$ . A stock solution of dithizone in chloroform containing 10 mg/ml will keep for a long period of time and is convenient for making the more dilute solution used in the test.

*Ammonia-Cyanide Mixture*. To 100 ml of 10% recrystallized phosphate free potassium cyanide in a 500 ml volumetric flask, add ammonium hydroxide equivalent to 19.1 g of ammonia and dilute to volume with distilled water.

#### Procedure

The limiting factor in the determination of small quantities of lead by the colorimetric dithizone method is the amount of contamination in the reagents. The importance of careful blank determinations must be especially stressed when quantities of lead in the order of 1 to 5 mcg are being determined. With special care in the purification of reagents by dithizone extraction and by use of carefully cleaned pyrex ware, including separators, it is possible to reduce the blank to 1 mcg or less. Due to lead bearing dust and vapors, it is necessary to expose blank determinations in the muffle furnace or on the steam bath for the same length of time as the sample is exposed and to use the same quantities of reagents for blank and determinations.

Lead is extracted from aqueous solution under standard conditions of volume and pH with a definite volume of dithizone solution at a standard concentration. The optimum pH is 9.5 to 10.0 while the volume and concentration of dithizone are 25 ml and 10 mg/liter respectively. This concentration ensures that excess dithizone is always present in the reaction mixture.

Lead is extracted by the chloroform as the red complex; uncombined green dithizone partitions between the aqueous and chloroform phases and thus modifies the color of the extract according to the relative quantities of lead complex and free dithizone. A series of colors from red to green may result depending on the amount of lead present. The colors produced with standard quantities of lead furnish the basis for quantitative determination of lead in unknown samples.

#### Standard

Into each of six 250 ml separatory funnels add 0, 1, 2, 3, 4, 5 ml aliquots of *Lead Standard Solution*.

Add enough 1% nitric acid to each to make 50 ml of solution. The acid should be added to the separatory funnel before the lead solution so that lead is not lost around the stopcock. Add 10 ml of ammonia-cyanide mixture and shake. The resultant pH is approximately 9.7.

Immediately add 25 ml of standard dithizone solution (10 mg/l) and shake vigorously for one minute. Allow to separate and filter the dithizone extract (lower layer) through specially prepared 9 cm filter paper into a 50 ml beaker.

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PART II

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Read the absorbance of the filter extracts at 510 nm in 1 cm cells. Correct for blank absorbance using the zero-added lead extract. Read the remaining five extracts vs the zero-added lead extract. Plot the absorbance at 510 nm against quantity of lead in extracts to obtain the standard curve.

**Sample**

For determination of lead in sample, take 20 ml aliquot of a previously prepared sample equivalent to 10 g per 100 ml and proceed as above with: "Add enough 1% nitric acid to each to make 50 ml of solution. . . ." If large quantities of zinc, manganese, cadmium, etc., are present, add sufficient 10% KCN to complex the interferences. Read the absorbance at 510 nm and determine the quantity of lead in the aliquot from the standard curve.

**Calculation**

$$\text{Lead (as ppm Pb)} = \frac{C \times F}{W \times V}$$

Where: C = conc of lead from standard curve, mcg  
F = dilution factor  
W = weight of sample  
V = volume (ml) of aliquot taken for analysis

**References**

1. Official Methods of Analysis—AOAC, 10th Edition, 1965, pp. 369–71
2. JAOAC 19 130 (1936)
3. *Ind. Eng. Chem., Anal. Ed.* 11 400 (1939)

\* \* \* \* \*

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C-T-F-A Method J 4-1

## **Asbestiform Amphibole Minerals in Cosmetic Talc**

**Part I: X-ray Diffraction Method**

**Part II: Optical Microscopy and Dispersion-Staining  
Method**

### **Introduction**

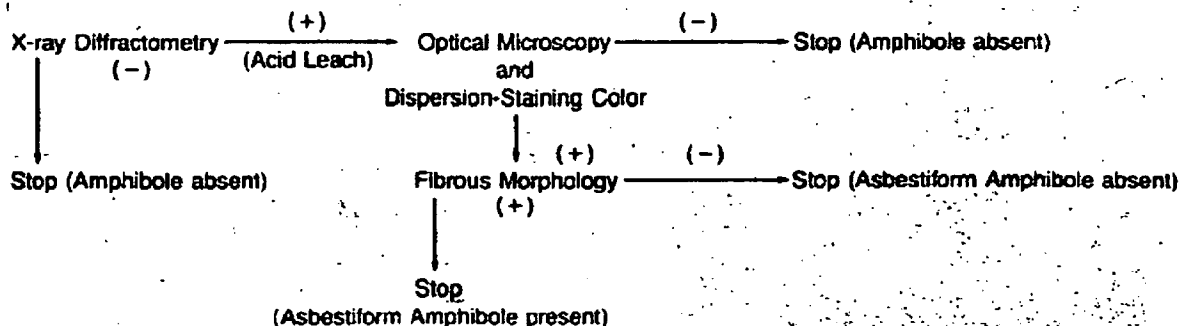
The method which has been adopted for the detection of amphibole minerals in cosmetic talc is the generally accepted method of x-ray diffraction. Methods which appear in the literature for the detection of fibrous amphibole, such as transmission electron microscopy with selected area diffraction<sup>1</sup> and electron microprobe,<sup>2</sup> have also been considered since they are capable of a lower level of detection than by x-ray diffraction. However, they have not been adopted since they suffer from the drawbacks, that the amount of material under examination is quite small (less than a microgram) and the time for analysis, expertise required, and expense of equipment eliminates them as routine methods.

The methodology presented is the most practical available, based on current technology. The use of Transmission Electron Microscopy with Selected Area Electron Diffraction offers greater sensitivity, but is not presented since it is unsuitable for normal quality control application.

Enrichment or concentration techniques using flotation cells have been tried as a means of improving the detection level; however, all efforts so far have been unsuccessful.

### **Principle**

The x-ray diffraction method is based upon the principle that when a crystalline material is placed in an x-ray beam, a portion of the x-rays are diffracted by each set of atomic planes within the crystal. The diffracted rays strike a scintillation counter as the sample is scanned through a prescribed angle with the resulting development of peaks corresponding to each interplanar distance ( $d$ ). A peak with  $d$  value in the range of 8.04 to 8.85Å for a sample talc is strong evidence for the presence of amphibole in that talc. The level of detection of amphibole by this method is 0.5% and above. The variability of detection is caused by such factors as age and manufacturer of x-ray diffractometers, sample homogeneity, specific amphibole mineral present, morphology of amphibole, particle size, preferred orientation, etc. For these reasons the level of detection should be reported for levels above 0.5%, since below this level the data has been found to be not reproducible. If a statistically significant peak is found of intensity equal to or greater than that obtained for the 0.5% standard in the  $d$  range for amphibole, described above, then the sample must be put through the following confirming scheme:



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**Part I: Amphibole Minerals by X-ray Diffractometry**

**Apparatus**

1. X-ray diffractometer, employing nickel-filtered copper K- $\alpha$  radiation, horizontal or vertical goniometer with variable scan speed capability, suitable talc pellet sample holder, variable speed recorder, electronic panel including ratemeter and variable attenuation and time constant settings
2. Hydraulic press, capable of attaining a pressure of 15,000 to 24,000 lb calculated on a 3" ram
3. Mortar and pestle or grinding mill (Note 1)
4. Waring Blendor,\* or equivalent blender
5. Spex Mixer/Mill,\* or equivalent mechanical mixer
6. Sieve, 325-mesh
7. Optical microscope (Note 2)
8. 1  $\frac{1}{2}$ " pellet press

**Reagents**

1. Standard talc sample, containing no detectable amphibole minerals
2. Standard tremolite sample, at least 80% pure
3. Denatured ethanol
4. Boric acid

**Procedure**

The procedure consists of slow-scanning, under previously determined conditions, a compressed pellet of the sample talc in the 11.0 to 10.0°2 $\theta$  (8.85 to 8.04 Å) region for the presence of an amphibole peak. There are times when it is difficult to discriminate a possible peak for amphibole over the background noise level. Should the presence of a small amphibole peak above the background "noise" be in question, it will be necessary to statistically evaluate the scan. A timer/scaler is required on the electronic panel of the x-ray diffractometer. In order for a peak to be statistically significant, the peak intensity must equal or exceed three standard deviations (3 $\sigma$ ) above the average background intensity (N):

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Where  $N + 3\sigma$  = minimum peak intensity  
 $N$  = average background count  
 $\sigma = N$

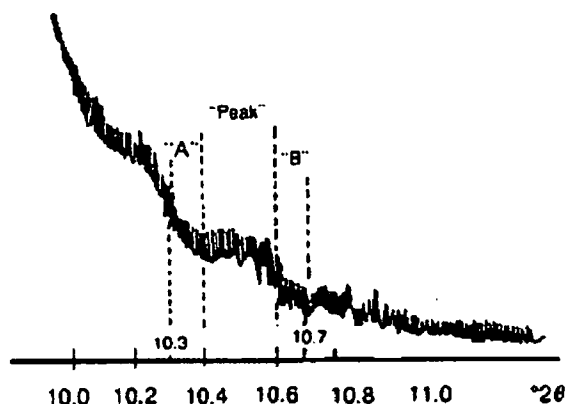


Figure 1.

Determine the region of the scan in question: in the Figure 1 scan, a peak appears to be present in the 10.40 to 10.60°2θ region.

Slow scan with cumulative pulse counting through the peak region three separate times and average the number of counts.

Determine a background count by scanning a region equal to  $\frac{1}{2}$  of the °2θ region covered by the peak, immediately, before and after the peak. The counting time for each of these background regions will equal  $\frac{1}{2}$  the total counting time used for the peak. Count each background region three times. Then average each region and add the two averages to obtain the background count (N).

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Example:

In Figure 1.

	Region (°2θ)	Time (sec.)
Peak .....	10.40 to 10.60	120
Background		
Region A .....	10.30 to 10.40	60
Region B .....	10.60 to 10.70	60

Peak		Background		Background	
10.40 to 10.60°2θ		Region A 10.30 to 10.40°2θ		Region B 10.60 to 10.70°2θ	
time secs.	counts	time secs.	counts	time secs.	counts
120	60,332	60	28,784	60	28,506
120	59,870	60	28,943	60	28,368
120	60,105	60	28,634	60	28,204
Average	60,102		28,787		28,359

$$N = 28,757 + 28,359 = 57,146$$

$$\sigma = \sqrt{57,146} = 239 \quad 3\sigma = 717$$

$$N + 3\sigma = 57,146 + 717 = 57,863$$

The actual number of counts obtained for the integrated peak intensity was 60,102; therefore, the "suspect" peak is statistically present in the scan.

**Standard Preparation**

Optimal instrument conditions must first be determined with the use of tremolite standards: 1.0%, 0.75%, 0.5% tremolite by weight, prepared in a standard talc which is free of interfering peaks in the 11.0 to 10.0°2θ region.

Weigh out appropriate amounts of standard talc and tremolite both of which have been ground to pass a 325-mesh sieve. Transfer to a Waring Blendor.\* Add 100 ml of ethanol to the blender and blend at low speed for 5 minutes.

Carefully transfer the contents of the blender, with repeated ethanol washings, into a large beaker. Evaporate the ethanol on a steam bath.

Shake the sample in a plastic vial for 5 minutes on a Spex Mixer/Mill\* to remove clumps and caked sample resulting from the evaporation of ethanol.

Determine by microscopy the homogeneity of the prepared standard previous to the x-ray diffraction analysis.

Press the homogeneous standard into a 1 1/4" pellet with a backing of boric acid. Transfer 2 (±0.2) g of standard to the die-holder and evenly distribute on a polished, scratch-free die. Distribute 4 (±0.2) g of boric acid evenly on the talc layer. Press the mixture into a pellet under conditions suitable for obtaining a smooth planar surface (for example, a pressure of 15,000 to 24,000 lb calculated on a 3" ram has been found to produce suitable pellets). The resulting pellet must have a talc face which is free of flaws; if not, the pellet must be discarded (Note 3). Prepare two acceptable pellets from each standard.

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**Sample Preparation**

Prepare two pellets from each sample in the manner described for the standard pellets. Make a qualitative scan from  $4$  to  $50^{\circ}2\theta$  on one of these pellets to ascertain the presence of amphibole above the 2% level or the presence of mineral impurities having interfering peaks in the  $11.0$  to  $10.0^{\circ}2\theta$  ( $8.85$  to  $8.04 \text{ \AA}$ ) region of the scan. The presence of such interference will eliminate use of the x-ray diffraction method for the sample, and one will have to proceed directly to the microscopical procedure.

**Instrumentation**

Instrumental variables are optimized on the 1% standard. Lower standards are then analyzed under the optimum conditions to determine the lower level of detection. Of major importance in obtaining maximum instrument sensitivity are a slow diffractometer speed combined with compatible recorder speed, and high attenuation combined with a statistically acceptable time constant on the ratemeter. Under appropriate instrumental conditions the peak obtained for the 0.5% standard should be detectable above background noise as shown in Figure 2.

Typical instrumental conditions employed for the Siemens Diffractometer (Model No. M386-XA4), and Counter and Recorder Unit (Type T) are:

Radiation:	Cu with $K_{\alpha}$ filter at 40 KV and 24 ma
Divergence slit:	$1^{\circ}$ Receiving slit: 0.2 mm
Goniometer speed:	$1/10^{\circ}2\theta/\text{minute}$
Recorder Speed:	300 mm/hour
Attenuation:	$1 \times 10^3$ impulses/second
Time constant:	$T(s) = 4$

Statistical error of 1.1% under these conditions

Rise Time = 0.18  
Attenuator = 20

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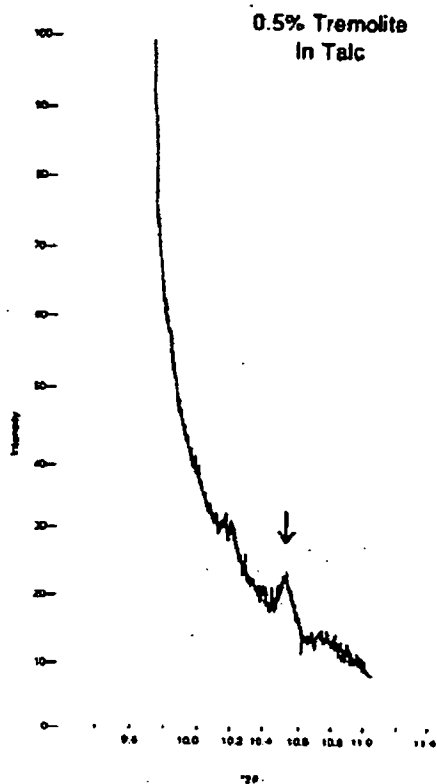


Figure 2

**X-Ray Diffraction Scans**

Place the standard or sample pellet in a suitable holder and slowly scan between 11.0 and 10.0°2θ. Then rotate the pellet 90° with respect to its original position in the goniometer and rescan between 11.0 and 10.0°2θ since pellet orientation may affect peak intensity. The presence of a reproducible peak (or peaks) is due to the presence of amphibole mineral (or minerals); the absence of peaks in this region indicates the absence of amphibole in the sample, within the limit of detection of this technique.

Report results as "None detected" or as "Detected approximately X% level," where "X" equals the level detected.

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**Part II: Asbestiform Amphibole Minerals by Optical Microscopy and Dispersion-Staining**

**Apparatus**

1. Polarizing microscope. Best results will be obtained if the instrument includes the following:
  - a. Individually centering objectives
  - b. Bertrand lens
  - c. High-intensity light source
  - d. Centering condenser/substage
2. Dispersion-staining device (Note 4)
3. Vacuum filtration equipment, including either a porcelain cone with glass fiber filter mat or a porous glass bottom cup

**Reagents**

1. Hydrochloric acid, 10% v/v
2. Cargille immersion liquid Series HD,  $n_D^{25} = 1.605$  (Note 5)

**Procedure**

**Acid Treatment**

Because of the interference caused by some carbonates (e.g., calcite) in the detection of asbestiform amphiboles in talc by optical microscopy/dispersion-staining, it is necessary to first remove these carbonates by a simple acid leaching procedure:

Weigh out 2 g of the talc into a 100 ml beaker. Add 25 ml of 10% v/v HCl slowly (to prevent excessive evolution of gas if carbonates are present) and heat, with occasional stirring on a steam bath for 30 minutes.

Filter with vacuum filtration equipment, and wash several times with hot water. Dry the talc.

**Optical Microscopy and Dispersion-Staining**

Carefully disperse 0.1 mg of talc in one drop of Cargille HD liquid,  $n_D^{25} = 1.605$ , and cover with a clean cover slip.

Examine the sample in the dispersion-staining central stop mode. The substage diaphragm should be almost completely closed, the field diaphragm may be partially closed to enhance color contrast, and the polarizer should be in position.

Tremolite, actinolite and presumably other amphibole minerals, under these conditions, will show the following dispersion-staining colors: yellow changing to blue with rotation of the sample relative to the polarizer or yellow changing to orange with rotation. The variation of the color change is due to the fact that the tremolite may lie in one of two positions relative to its principal optical orientation.

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Examine the sample for asbestiform fibrous amphibole minerals.

In order for an amphibole mineral to be considered asbestiform fibrous it must meet the following OSHA definition (Reference 4).

1. Particles must appear to be fibrous rather than as crystals or silvers.
2. The maximum diameter of a fiber to be counted is 3 microns.
3. The maximum length of a fiber to be counted is 30 microns.
4. The length to width ratio must be 5 or more to 1, that is, 5 times or more longer than wide.
5. The separate or individual fibers must contain fibrils or the "bundle of sticks" effect, unless they are at a nondivisible stage. A fibril cannot be subdivided and would be counted, if it meets the other criteria. The length to width ratio of 5 or more to 1 is not meant to imply that other particles are not hazardous.

Report results as "Asbestiform Amphibole Present" or as "Asbestiform Amphibole Absent."

It is imperative that both dispersion-staining color and fibrous morphology criteria be satisfied before identifying a particle as asbestiform amphibole, since other substances may show colors similar to those described.

**Notes**

1. Talc to be analyzed and the tremolite used to prepare standard samples must be finer than 325 mesh (maximum particle size of 44 microns). The Tekmar Analytical Mill (Model A-10) is available from:

Tekmar Company  
P.O. Box 37202  
Cincinnati, Ohio 45222

2. It is important that the homogeneity of the prepared talc-tremolite standard samples be verified by optical microscopy.
3. This requirement is critical since excessive surface scatter will cause abnormally high background count.
4. The only commercially available dispersion-staining device is available from:

Walter C. McCrone Associates, Inc.  
2820 South Michigan Avenue  
Chicago, Illinois 60616

5. Available from:

R.P. Cargille Laboratories, Inc.  
Cedar Grove, New Jersey 07009

—or from laboratory suppliers.

**References**

1. Rohl, A. N., Langer, A. M., *Environmental Health Perspectives* 9, 95 (1974)
2. Rubin, I. B., Maggiore, C. J., *Environmental Health Perspectives* 9, 81 (1974)
3. L. S. Birks, *X-Ray Spectrochemical Analysis*, pages 54–55, Interscience Publishers (1959)
4. "Tremolite and Talc." U. S. Department of Labor, Occupational Safety and Health Administration, Field Information Memorandum #74-92, November 21, 1974

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J&J-0039106

## **Free Crystalline Silica (Quartz) in Talc** **(DTA Method)**

### **Principle**

Differential thermal analysis (DTA) involves the measurement of thermal reactions which are induced in a sample as it is being heated at a constant rate. First-order thermal transitions are denoted as endothermic or exothermic depending upon whether the process is accompanied by the absorption or release of energy in the form of heat. The sample holder includes a reference thermocouple and a differential thermocouple, which detect thermal reactions by continuously monitoring any difference in temperature between the sample material and a thermally inert reference substance (calcined alumina) contained in another cavity or sample dish of the holder.

The DTA method permits the unequivocal detection of quartz (free crystalline silica) in talc at a 0.5 to 1.0% w/w minimum detectable level. The method utilizes the thermal transition representing the reversible alpha to beta crystal inversion of quartz at 573 °C. On heating, the latent heat of inversion gives rise to an endothermic reaction; on cooling, an exothermic transition is obtained. The talc is first calcined at approximately 800 °C for the purpose of inducing irreversible thermal transitions attributable to mineral impurities. The cooling curve then shows a flat baseline, thus improving detectability for quartz. Studies have shown that the intensity of this thermal peak is affected by the particle size distribution of the quartz. Therefore, DTA is not recommended for the quantitative determination of quartz.

### **Apparatus**

1. Differential Thermal Analyzer, including either a high temperature powder sample holder of nickel or stainless steel construction with exposed-loop differential thermocouple (such as Platinel II) or sample holder employing a ring type differential thermocouple with platinum dishes, with equipment to heat to at least 800 °C
2. Spex Mixer/Mill,\* or equivalent mechanical mixer with plastic vial and plastic ball
3. Sieve, 325-mesh
4. Mortar and pestle, or grinding mill

### **Reagents**

1. Standard talc sample, containing no detectable quartz
2. Standard quartz sample, at least 95% pure

### **Procedure**

#### **Standard Preparation**

Grind standard talc and quartz samples to pass a 325-mesh sieve. This will give a particle size distribution of 0 to 44 µm.

Weigh appropriate amounts of the ground standard talc and quartz into a plastic vial to prepare 1.0% w/w quartz-talc standard. Mix with a plastic ball approximately 10 minutes in Spex Mixer/Mill.\*

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**Instrumental Parameters**

Experimental conditions for a typical DTA unit (Stone Model RC-202C or LA-XYH) are as follows:

Sample holder: high temperature powder type, of nickel or stainless steel construction (Model SH-8BE2), with exposed-loop differential thermocouple (Platinel II), Imbedded reference thermocouple (Platinel II or Chromel/Alumel)

Temperature range: ambient to 800 °C

Sample: 130 to 150 mg talc, using a loose, consistent packing technique

Reference material: alumina, ground to pass 325-mesh sieve

Atmosphere: static air

Heating rate: 10 °C/min

Sensitivity: 40  $\mu$ volls full scale

Furnace: LTF, water-cooled to 1200 °C

Instrumental parameters must be determined for a particular differential thermal analyzer such that 0.5 to 1.0% w/w quartz in talc may be detected in the cooling curve subsequent to calcining the talc at 800 °C. Once these experimental parameters have been determined, they must not be altered during the analysis of talc samples in order to assure instrument sensitivity.

It is emphasized that reproducibility of the method is based on standardizing the experimental conditions.

Run the prepared quartz-talc standard to optimize sensitivity of the instrument.

If the quartz inversion endotherm at 573 °C is masked by thermal transitions of mineral impurities in the talc, it is necessary to rely on a cooling curve for quartz detection. If there is no provision on the DTA instrument for programmed cooling, heat the talc to 800 °C, cool to room temperature, and record the thermogram on reheating for detection of quartz.

Grind talc or talc ore samples to pass 325-mesh sieve to give particle size distribution comparable to standard sample. Run the prepared talc sample under the same identical conditions as the standard. Detect the quartz by means of the crystal inversion at 573 °C on heating or cooling (Notes 1 & 2).

Report results as "None detected," or as "Equal to or greater than 0.5 to 1.0% w/w."

**Notes**

1. The value of the DTA method lies in the specificity of the determination over that attainable by x-ray diffraction in the silicate mineral matrix. No other mineral has been reported in the literature to have a thermal transition peak at 573 °C. Semi-quantitative DTA is feasible only under fixed experimental conditions and with some knowledge of quartz particle size.
2. If semi-quantitative estimation of the quartz content is desired, this may be achieved under fixed experimental parameters by comparison of quartz peak intensities obtained for additional quartz-talc standards (Figure 1). The particle size distribution of the quartz in talc samples and standards must be as consistent as possible.

\* \* \* \* \*

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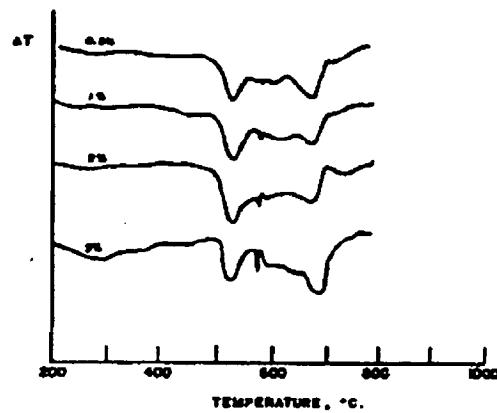


Figure 1

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C-T-F-A Method J 6-1

## **Free Crystalline Silica (Quartz) in Talc** **(X-ray Diffraction Method)**

### **Principle**

X-ray diffraction is a convenient method for determining the presence of crystalline impurities, such as free crystalline silica (quartz), in talc. It is necessary that at least the three strongest diffraction lines of quartz be present in the x-ray pattern to confirm the presence of quartz in the talc: 3.34, 4.26, 1.82 dÅ.

It has been experimentally determined that compression of a talc sample containing quartz into a pellet results in an x-ray diffraction pattern having more intense quartz peaks than the pattern obtained from a packed-powder sample of the same talc. Using this pressed pellet technique, it is possible to detect the three strongest quartz peaks at a minimum level of 2% w/w.

### **Apparatus**

1. X-ray diffractometer with nickel-filtered CuK $\alpha$  radiation and a suitable 1  $\frac{1}{2}$ " circular sample holder for the pelletized sample
2. Hydraulic press capable of maintaining from 15,000 to 24,000 lb (as calculated on a 3" ram)
3. 1  $\frac{1}{2}$ " pellet press

### **Reagent**

Boric acid

### **Procedure**

#### **Sample Preparation**

Transfer approximately 2 g of sample talc to the die-holder and distribute evenly on a polished, scratch free die; then distribute approximately 4 g of boric acid on top of the talc layer. Compress the two layers into a pellet whose talc face is free of flaws (Note).

### **Instrumentation**

Scan the sample pellet by x-ray through the three analytical quartz regions under instrumental conditions which have previously been determined sensitive to the 2% w/w quartz level.

As an example, the following conditions have been employed on the Siemens Diffractometer (Model No. M386-X-A4) and Siemens Counter and Recorder (Type T):

Radiation: Cu with K $\beta$  filter at 40 KV and 24 ma  
Divergence slit:  $\frac{1}{2}$ " Receiving slit: 0.2 mm  
Goniometer speed:  $\frac{1}{2}$ " 2 $\theta$ /minute  
Recorder speed: 600 mm/hour  
Attenuation =  $1 \times 10^3$  impulses/second  
Time constant, T[S] = 1  
Rise time = 0.18 Attenuator = 20

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**X-Ray Diffraction Scans**

First scan the sample pellet from  $25.5$  to  $27.5^\circ 2\theta$  for the presence of a peak at  $26.7 (\pm 0.1)^\circ 2\theta$ . If no peak is detected, quartz is absent at the lower limit of detection.

If a peak is observed in the above scan, then scan the pellet through the  $19.5$  to  $21.5^\circ 2\theta$  region for a quartz reflection at  $20.8 (\pm 0.1)^\circ 2\theta$ . The absence of a peak confirms the absence of quartz at or above 2% w/w.

If both peaks are observed, scan the sample from  $49.0$  to  $51.0^\circ 2\theta$  for the presence of a peak at  $50.1 (\pm 0.2)^\circ 2\theta$ .

The presence of all three peaks confirms that the talc contains quartz at a level equal to or greater than 2% w/w.

Report results as "None detected," or as "Equal to or greater than 2% w/w."

**Note**

The amount of pressure necessary for a good talc face may vary from talc to talc and can only be determined experimentally.

\* \* \* \* \*

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